

CLAIMS

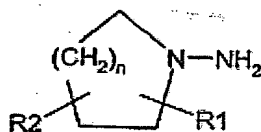
1. Method for synthesizing derivatives of exocyclic cycloalkyl-hydrazines and heterocycloalkyl-hydrazines, characterized in that it comprises the following successive steps:

a) synthesizing the derivative of exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine in a suitable reactor by causing a monochloramine to react with a heterocyclic amine in an alkaline medium at a temperature of between 30 and 60°C; then

b) demixing the solution obtained at step a) into an organic phase and an aqueous phase through the addition of anhydrous sodium hydroxide under cooling so that the temperature does not exceed the boiling point of the compounds; and

c) optionally, isolating the derivative of exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine by distilling the organic phase obtained.

2. Method as in claim 1, characterized in that the derivative of exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine has the formula (I):



in which one of the carbon atoms of the cycle is optionally replaced by a heteroatom chosen from among a nitrogen or oxygen atom, R1 and R2 identical or different represent a hydrogen atom or a C₁-C₆ alkyl radical, or R1 and R2 together form a C₃-C₈ cycloalkyl radical and n equals 1 to 3.

3. Method as in claim 1 or 2, characterized in that at step a) the heterocyclic amine / monochloramine molar ratio lies between 4 and 10.

5 4. Method as in any of the preceding claims, characterized in that the reactor of step a) is placed in an inert atmosphere.

10 5. Method as in any of the preceding claims, characterized in that prior to step a), the monochloramine is alkalised in a mixer through the addition of a solution of sodium hydroxide so that the weight percentage of sodium hydroxide lies between 2 and 6 %.

15 6. Method as in claim 5, characterized in that the mixer is maintained at a temperature of between -10 and 5°C.

20 7. Method as in any of the preceding claims, characterized in that the quantity of anhydrous sodium hydroxide added at step b) is such that the weight percentage of sodium hydroxide is between 10 and 35 %.

25 8. Method as in any of the preceding claims, characterized in that the heterocyclic amine is added at step a) in the form of anhydrous heterocyclic amine.

9. Method as in claim 8, characterized in that step c) comprises the following successive steps:

30 i) isolating the heterocyclic amine which has not reacted and a concentrated solution of the exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine derivative by distillation of the organic phase obtained after step b); then

35 ii) optionally rectifying, by distillation under reduced pressure, said concentrated solution of the derivative of exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine.

10. Method as in any of claims 1 to 7, characterized in that the heterocyclic amine is added at step a) in the form of a concentrated aqueous solution of heterocyclic amine, optionally in the form of a water-heterocyclic amine azeotrope.

11. Method as in claim 10, characterized in that after step a) and prior to step b), the method comprises the following steps:

10 i') removing the ammonia present in the solution obtained after step a) by stripping; then

ii') isolating a solution containing the formed derivative of exocyclic cycloalkyl-hydrazine or heterocycloalkyl-hydrazine and an aqueous, optionally azeotropic, solution of heterocyclic amine which has not reacted, by distilling the solution obtained after step i') at a temperature of between 50 and 180°C; and

15 iii') reinjecting into the reactor of step a) said aqueous, optionally azeotropic, solution of heterocyclic amine obtained after step ii').

12. Method as in any of the preceding claims, characterized in that the monochloramine is prepared using a method comprising the following successive steps:

25 α) preparing an aqueous solution of sodium hypochlorite whose chlorometric degree is between 36 and 100°, optionally by diluting a hypochlorite solution with between 100 and 120° chlorometric degrees; then

30 β) causing a solution of ammonium hydroxide and ammonium chloride to react with the aqueous solution of sodium hypochlorite obtained after step a), in a low alkaline medium, at a temperature lying between -15 and -7°C to form said monochloramine.

35 13. Method as in claim 12, characterized in that the molar ratio of ammonium hydroxide and ammonium chloride

solution / aqueous solution of sodium hypochlorite is between 2.5 and 3.

14. Method as in claim 12 or 13, characterized in that
5 the molar ratio of ammonium chloride/ammonium hydroxide is between 0.1 and 1.75, advantageously it is approximately 0.65.